EFFECT OF COPPER ON THE PROPERTIES OF AUSTEMPERED DUCTILE IRON CASTINGS

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE

REQUIREMENT FOR THE DEGREE OF

Bachelor of Technology

in

Metallurgical and Materials Engineering

By

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2012

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CERTIFICATE

This is to certify that the thesis entitled, **"EFFECT OF COPPER ON THE PROPERTIES OF AUSTEMPERED DUCTILE IRON CASTINGS"** submitted by **Mr. GOURAHARI BEHERA** and **Mr. SOUMYA RANJAN SOHALA** in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Metallurgical and Materials Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

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ABSTRACT

Two types of ductile iron has been taken for the present investigation. Both austempering time and temperature are considered as the main variables for structure property correlation of Austempered Ductile Iron. The two types of spheroidal graphite iron (differ by copper percentage) were austempered at four differing austempering temperatures viz. 250^{o}C, 300^{o}C, 350^{o}C and 400^{o}C for 60min, 90 min and 120 min respectively. The effect of austempering variables on the mechanical properties of spheroidal graphite iron was investigated as a function of austempering time and temperature. The cooling rate and the quenching techniques followed in the present study plays an important role for the property development of spheroidal graphite iron. The tensile properties have been correlated with the graphite morphology for both grades of ADI. SEM micrographs have been taken from fracture surfaces of the tensile specimen under different austempering conditions. It has been found from the result that ADI having the alloying element (copper) achieved significant mechanical properties as compared to ADI without having copper throughout the different austempering processes adopted in this study.

Keywords: spheroidal graphite iron; ADI; austempering time and temperature; SEM analysis.

CHAPTER 1

INTRODUCTION

INTRODUCTION

In recent years, light weight,durable and cost effective materials are in demand for energy saving. Researchers are trying to formulate new materials which may solve the purpose. They found ductile iron as one such material.They are doing researchon this material and have focused on possible improvements of mechanical properties by appropriate heat treatment processes and also by alloying, which can enhance its properties.

Ductile iron when subjected to an isothermal heat treatment process known as "austempering" produces Austempered Ductile Iron (ADI). It has better properties than ductile iron.

After austempering ofthe ductile cast iron, the resulting microstructure is known as "Ausferrite", which consists of fine acicular ferrite with carbon enriched stabilized austenite [2]. The new microstructure results with capability superior to many ferrous and aluminium alloys. As compared with pearlitic, ferritic or martensitic structures, ausferrite exhibits twice the strength for a given level of ductility formed by conventional heat treatment processes.

The mechanical properties of the austempered ductile iron depend on the ausferrite microstructure. The austempered matrix offers better tensile strength to ductility ratio than is possible with any other grade of ductile iron [1]. Various combinations of properties can be obtained from austempered ductile iron because of the ausferrite microstructure which depends on heat treatment conditions and alloyed elements.

In present research work, the effect of copper alloying as well as the effect of heat treatment parameters like austempering temperature and austempering time on microstructure and properties of the ductile iron were studied.

CHAPTER 2

LITERATURE REVIEW

LITERATURE REVIEW

Alloys of iron and carbon such as steels having greater amount of carbon are basically called Cast Iron. The carbon content of it ranges between 2 and 6.67%. Due to high carbon content it is very brittle in nature and most commercially manufactured cast iron ranges from 2.5 and 4% .Ductility is also very low. As cast iron melted very easily and as they are very brittle in nature,casting is the only process for the manufacture of complicated shapes. Controlling the alloying addition, good foundry practice and appropriate heat treatment, the properties vary over a wide range. The shape and distribution of free carbon particles greatly influenced the physical properties of cast iron. Malleable cast iron, gray cast iron, white cast iron, nodular cast iron and alloyed cast iron are the different type of cast irons [3].

2.1. Ductile iron:

Ductile iron or nodular iron or spheroidal graphite cast iron is a type of cast iron where graphite is present in the form of nodules, tiny balls or spheroids [3]. It derives its name from the fact that in the as-cast structure it exhibits measurable ductility. Other types of cast iron do not exhibit this much of ductility. Based on the matrix present SG iron may be classified into different types namely, ferritic, pearlitic, martensitic and austenitic. Depending on the cooling rate the matrix may vary from a soft ductile ferritic structure through a hard and higher strength pearlitic structure to an austenitic structure. One of the most fascinating feature of ductile iron is that the tensile elongation is as high as 17% which is not comparable to other types of cast iron[4].

2.1.1. Composition of SG Iron:

In unalloyed ductile iron the contents of various elements ranges as follows:

Carbon	3.0 wt. % to 4.0 wt. %
Silicon	1.8 wt. % to 2.8 wt. %
Manganese	0.1 wt.% to 0.5 wt.%
Phosphorous	0.005 wt.% to 0.03 wt.%

Sulphur should be maximum 0.015 wt.%[5,6]

Carbon and silicon are the main elements for the composition of common cast iron. The amount of graphite or Fe_3C depends on the carbon content and increases as the carbon content increases. The graphitization potential of iron and castability depends on high carbon and silicon content. Typically, the Mg varies from 0.03% for ferrule irons to as high as 0.06% for pearlitic irons. Phosphorus and sulphur are also present in the composition. They can be as high as 0.15% for low-quality iron and are considerably less for high-quality iron, such as ductile iron or compacted graphite iron. The graphite morphology of ductile iron greatly depends on the chemical composition. Optimum amounts of carbon and silicon can prevents graphite floatation, high impact transition temperature, excessive shrinkage and high chilling tendency. Minor elements can also alter the structure significantly in terms of graphite morphology, chilling tendency and matrix structure.[9]

2.1.2 Production of SG iron:

1. Desulphurization: Sulphur has deleterious effects on the properties of SG Iron as the growth of graphite flakes depends on the content of sulphur. The sulphur content in SG Iron should be as low as possible (<0.015%). If the sulphur content exceeds this limit than desulphurization is done during melting by adding desulphurising agent like soda ash or calcium carbide.

2. Nodulising: Magnesium on reacting with oxygen form highly stable MgO which floats on the surface and can be skimmed off easily and the oxygen content is reduced from typical levels of 90-135ppm to about 15-35ppm. Magnesium on reacting with sulphur produces less stable Magnesium sulphide. Solubility of Mg is less in the metal and it is volatile, so a chance of reverse reaction is there if losses are not taken care of. For deoxidation Si is also added. Cerium forms stable oxides and sulphides with O and S respectively and is less volatile than Mg. Mg is added when the temperature of the melt is at 1500˚C but Mg vaporizes at 1100˚C. Mg being lighter floats on the bath and being reactive burn off at the surface. Mg is generally added as Ni-Si-Mg, Ni-Mg alloy or magnesium coke to reduce the violence of the chemical reaction and to have saving in Mg. After nodulising treatment, inoculants like Mg have their spherodising effect on the graphite structure so that graphite nodules can be formed.

3. Inoculation: For precipitation and subsequent growth of graphite at number of points inoculation does the purpose. In this process small amounts of inoculants are added to the molten metal either just before or during pouring. High levels promote graphite structure while low levels result in the formation of either mottled structure or white irons. A high level of nucleation increases as cooling rate increases. Along with inoculants Mg is a powerful carbide promoter and as a result, compared with the gray irons, there is a far greater tendency for ductile irons to solidify with white or mottled structure. To suppress the formation of chill and mottle, ductile irons is inoculated. Also it maintains a good nodule shape and also high nodule numbers. All silicon based inoculant are very effective. Among them the most widely used is foundry grade ferrosilicon, containing about 75% silicon. The inoculating effect produced initially increases as the amount of inoculants is increased, but the effect soon begins to level off. After that a situation is reached where the extra inoculating benefit obtained is too small to justify for the increased addition.[4]

4. Solidification:For solidification of SG iron undercooling is required. The graphite nuclei grow slowly and are then is surrounded by the austenite. At eutectic temperature the combination of austenite and graphite corresponds to the eutectic point. Supersaturation of Austenite with carbon takes place and on cooling a new equilibrium is established at the graphite-austenite interface. Towards the graphite nodule the excess of carbon diffuses and precipitates out.

Ductile iron or nodular iron is made by treating the liquid metal with magnesium or cerium before tapping followed by proper inoculation. This accelerates the precipitation of graphite in the form of spherical shapes precipitate uniformly in the matrix. This iron is ductile in nature and can be used in critical application for different components or machinery part in the industries. Wide range of ductile iron castings now days have been used in windmill parts. Some of examples are here under: Blade adapter,axle pin,rotor hub, bearing housing, brackets,valves,water pipes,nuclear fuel container. It is already reported that, as per availability and low cost Mg are used for the production of ductile iron irrespective of Ce. Ductile iron is prepared by taking low P pig iron, coconut charcoal,steel scrap,foundry return and required ferro alloys. These materials are known as charging materials. For melting, induction furnace is used which gives best result as per foundry procedure and practices. Materials are charged in the induction furnace as per standard procedure to get the optimum

chemical composition and poured in a Furan resin sand mould for casting. The tapping temperature basically maintained 1450±10^{o}C. And the pouring temperature was maintained at 1380±10^{o}C. From tapping to pouring the time is so adjusted(i.e. no delay in delivery of liquid metal) in order to avoid formation of carbide and lack of nucleation sites. Carbon equivalent plays an important role for structural property determination of ductile iron. It is reported that CE should be greater than 3.9(should be in the range of 3.9 to 4.5)

$$CE = C\% + \frac{1}{3}\ Si\ \%$$

Quality index has been made to compare the mechanical properties of SG iron. The formula used for this is

$$Q = \frac{[TensileStrength.ksi]sq * [Elongation\%]}{1000}$$

Where Q is a constant. The larger value of Q indicates a combination of higher strength and elongation which results higher performance in the material.

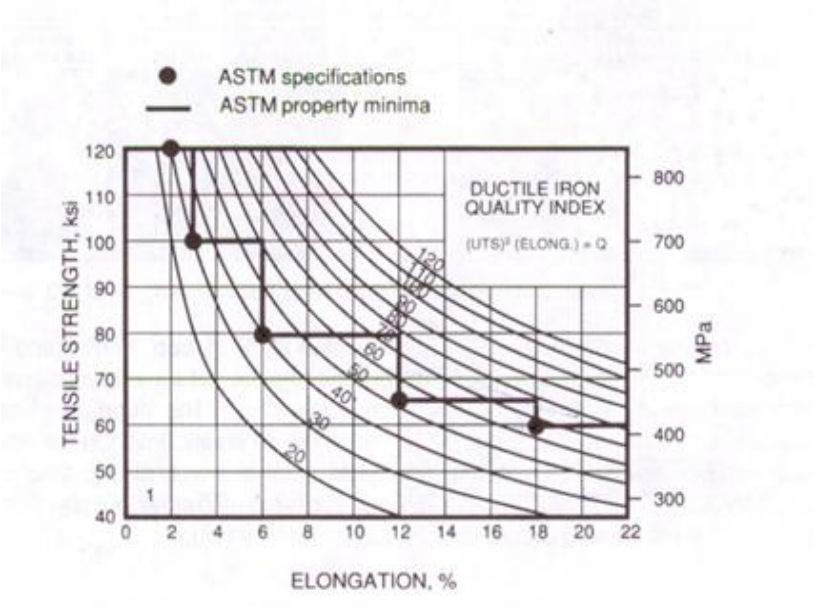

Fig 1. ASTM Quality Index graph for ductile iron

2.1.3. Properties of SG iron:

Depending on the grade of SG Iron its properties can vary. The tensile strength can vary from 400 MPa for ferritic grades to as high as 1300 MPa for austempered ductile iron grades. The yield strength can also vary over the range of 250 MPa to 800 MPa. The elongation can sometimes be as high as 25%, only for ferritic grades. High fluidity enables the castability of SG Iron. Also good machinability properties because of the graphite present which makes chip formation easier. And also ductile iron is highly corrosion resistant.[7,8]

2.1.4. Structure

The microstructure of ductile iron constitutes of: the morphological forms taken by carbon and the metal matrix in which the carbon and/or carbide particles are dispersed. The following important microstructural components are found in ductile iron.

2.1.4.1.Graphite:

Graphite is the most stable form of pure carbon in cast iron.

Some of the physical properties of graphite are as follows:

- low density
- low hardness
- high thermal conductivity
- Lubricant.

In cast iron graphite shape may vary from flake to spherical. In ductile iron all of its graphite occurs in spheroidal form microscopically and it constitutes about 10% by volume of ductile iron.[10]

2.1.4.2. Ferrite:

This is an interstitial solid solution of carbon in Alpha Iron and also the purest iron phase in a cast iron. Ferrite offers lower strength and hardness, but it is very ductile in nature which gives high ductility and toughness. Austempered Ductile Iron (ADI) contain acicular ferrite (very fine grained), which provides a good combination of strength with ductility and toughness. In ferritic ductile iron the strength properties are generally increased by the elements, which go into the solution. [10]

2.1.4.3. **Pearlite:**

Pearlite is a eutectoid mixture of cementite and ferrite produced by the eutectoid reaction. It provides higher strength but at the expense of reduction in the ductility which meets the requirements of many engineering applications.[10]

2.1.4.4**Martensite:**

It is a supersaturated solid solution of carbon in iron produced by rapid cooling or "quenching". It is very hard and brittle but ductility is very low. To increase the ductility it is normally "tempered" i.e. heat treated which reduces the carbon content by the precipitation of carbides.[10]

2.1.4.5. **Austenite:**

It is a high temperature phase consists of carbon dissolved in iron. Nickel stabilizes the austenitic irons and the amount of Ni should be in the range of 18 to 36%.[11]In austempered ductile irons, austenite is produced by a combination of

- Rapid cooling, suppress the pearlite formation.
- Supersaturationof carbon during austempering, depresses the start of the transformation of the austenite to martensite far below room temperature.

The austenite matrix provides

- ductility and toughness at all temperatures
- corrosion resistance
- And good high temperature properties, especially under thermal cycling conditions.[10]

2.1.4.6. **Bainite:**

Bainite is a mixture of ferrite and carbide (Fe_3C, $Fe_{2.4}C$) produced by heat treatment or alloying[10]

2.1.5. Types of ductile iron

The different types of ductile iron are as follows:

2.1.5.1. Ferritic Ductile Iron

Ferritic ductile iron is formed when graphite spheroids are present in a matrix of ferrite. It provides an iron with good ductility and impact resistance with a tensile and yield strength equivalent to low carbon steel. It can be produced as-cast and an annealing heat treatment may be given to assure maximum ductility and low temperature toughness[10, 12]

2.1.5.2. Ferrito- Pearlitic Ductile Iron

These are the most common grade of ductile iron where the graphite spheroids are present in a matrix containing both ferrite and pearlite. It is produced as-cast and its properties are intermediate between ferritic and pearlitic grades along with good machinability and low production costs[10, 12]

2.1.5.3. Pearlitic Ductile Iron

In these type of ductile iron the graphite spheroids are present in a matrix of pearlite which results in high strength, good wear resistance, and moderate ductility and impact resistant. It also shows good machinability [10, 12]

2.1.5.4. Martensitic Ductile iron

The graphite spheroids are present in a matrix of martensite. Sufficient alloy additions to prevent pearlite formation, and a heat treatment (quench and tempered) produces this type of ductile iron.This type of ductile iron develops very high strength and wear resistance but with lower levels of ductility due to tempered martensite matrix. [10, 12]

2.1.5.5. Austenitic Ductile Iron

When the iron is alloyed to produce an austenitic matrix this type of ductile iron is produced. It offers

- good corrosion and oxidation resistance
- good strength and dimensional stability at elevated temperatures

2.1.5.6. Austempered Ductile iron (ADI)

ADI is the most recent one added to the ductile iron family produced by austempering of the conventional ductile iron. It is nearly twice as strong as pearlitic ductile iron along with high elongation and toughness. This combination of properties provides a material with superior wear resistance and fatigue strength.[10,12]

2.1.6. Various grade of S.G. irons

These grades are accepted as per international norms.

TABLE 1:International standard ISO 1083 : 1987

Grade	Tensile Strength (N/mm^2)	0.2 % Proof Strength (N/mm^2)	Elongation (%)
900-2	900	600	2
800-2	800	480	2
700-2	700	420	2
600-3	600	370	3
500-7	500	320	7
450-10	450	310	10
400-15	400	250	15
400-18	400	250	18
400-18L	400	250	18
350-22	350	220	22
350-22L	350	220	22

2.1.7. Factors that affect the properties of the ductile iron:

Ductile iron exhibits a good combination of strength and ductility its huge application in heavy engineering industries. For its typical mechanical properties Some important constituents are listed.

2.1.7. 1. Effect of graphite shape

The graphite shape plays a significant role in determining the properties of ductile iron. The nodularity affects the mechanical properties of ductile iron. When nodularity is decreased i.e. the nodules become elongated, but do not become sharp or "spiky", yield strength decreases by 10 % and tensile strength by 15% when nodularity is reduced to 30%.[10]

2.1.7. 2 Effect of Nodule Count

Nodule Count is expressed as the number of graphite nodules/mm^2. High nodule count ensures good metallurgical quality, but excess nodule count may result in the degradation of the properties of ductile iron. Some of the effects of nodule count on microstructure are as follows:

- The pearlitic content of as-cast ductile iron decreases with increase in nodule count.
- It alsoaffects the carbide content and with increasing nodule count the volume fractions of chill carbides or segregation carbides decreases which ultimately improves ductility and machinability.
- Increasing nodule count produces more homogeneous and finer microstructure.
- Nodule count is inversely proportional to the graphile nodule size. Increase in nodule count decreases the graphite nodule size.

Nodularity and nodule count also can affect the mechanical properties and it depends upon the factor as follows.

- Inoculation fading.
- Mg recovery and Mg treatment temperature
- Pouring temperature and cooling rate [10]

2.1.7. 3. Effect of Matrix

In Ductile Iron the mechanical properties are determined primarily by the constituents of matrix and their hardness. The common grades of ductile iron has matrix consists of ferrite and/or pearlite. Ferrite in ductile Iron offers low strength and hardness but

high ductility, toughness and good machinability. Whereas Pearlite is an intimate mixture of lamellar cementite in ferrite matrix.Pearlite provides high strength and hardness but lower ductility. Therefore the mechanical properties of ferritic/pearlitic Ductile Irons aredetermined by the ratio of ferrite to pearlite in the matrix. By controlling the composition of the iron and the cooling rate of the casting, the ratio is controlled. By Annealing and Normalising the ratio can be controlled, as annealing produces ferritic and normalising produces pearlitic matrix.[10].

These irons are cast after treatment of the molten metal with a small quantity of magnesium to change the graphite forms a flake to nodular or spheriodal form.

Graphite nodules are small and constitute only small planes of weakness in the steel – like matrix, these being about one two – hundredth the size of those occurring when flake graphite is present. Because of this, and also because the graphite is spheroidal, stress concentrations round the graphite are small, and ductile irons have mechanical properties which relate directly to the strain and ductility of the matrix present as is the case for steels. Unlike gray cast irons, which generally fail with less than about one percentage elongation, ductile iron can deform plastically before fracture and have elongation of 2 –25 %. Strengths generally decrease as the elongation increases. When high – strength matrix structures are obtained by heat treatment, such as normalizing, hardening and tempering, and austempering, it is possible to produce irons with both increased strength and relatively high elongations.

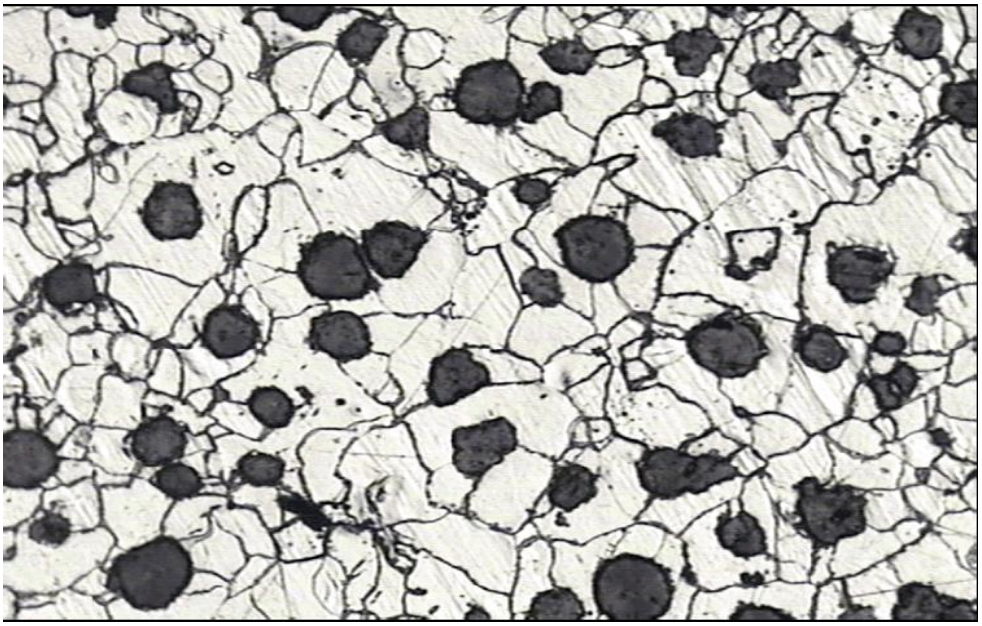

Fig 2: Microstructure of ferritic ductile iron(100X,2 % Nital etched)

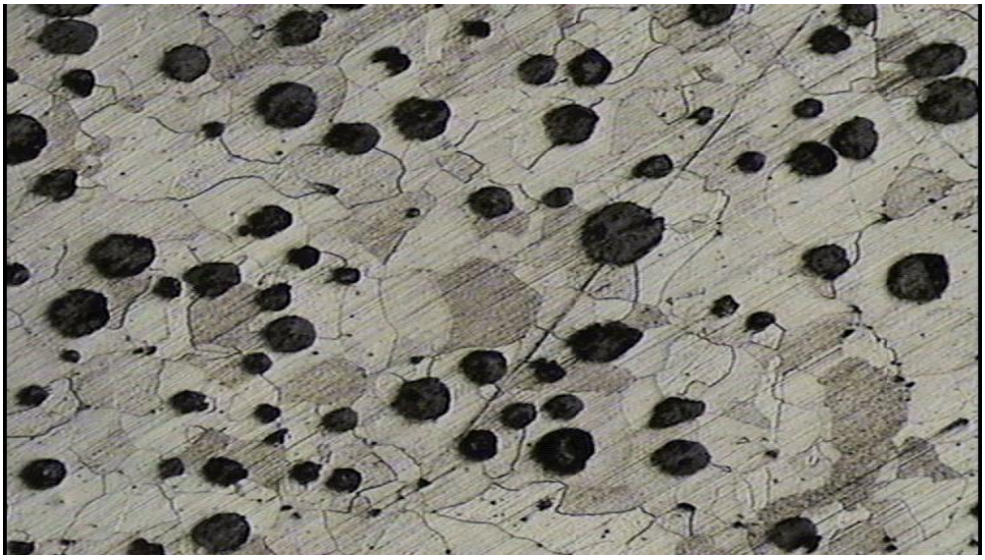

Fig 3: Microstructure of pearlitic ductile iron(100X,2 % Nital etched).

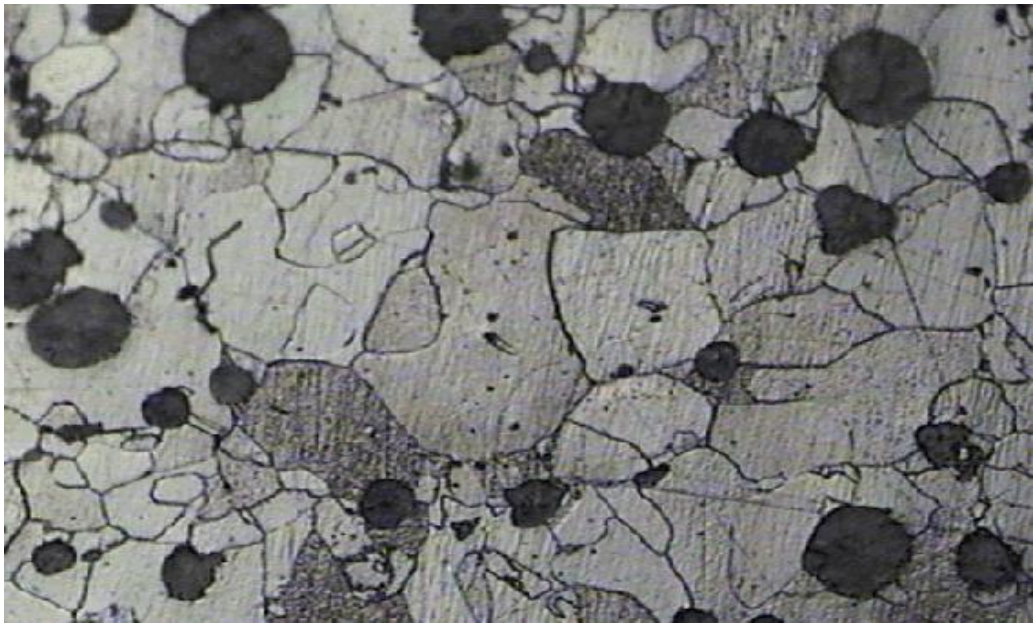

Fig 4:Microstructure of Ferrito-pearlitic ductile iron (100X,2 % Nital etched**)**

2.1.7. 4. Effect of silicon

At elevated temperature for stabilizing ferritic matrix in ductile iron silicon is normally used. It forms a silicon-rich surface layer which helps in inhibiting oxidation. Silicon generally raises the critical temperature of thetransformation of ferrite to austenite. Above this critical temperature the expansion and contraction can cause distortion and cracking of the casting and surface oxide layer respectively, thus reducing oxidation resistance. Due to high ferritizing tendency of the silicon, the formation of carbides and pearlite are prevented and stabilizes the matrix. Silicon offer protection against oxidation and it increases with increasing silicon content. [14]

2.1.7. 5. Effect of molybdenum

Molybdenum helps in increasing the creep and stress-rupture properties of steels. On adding 0.5 % molybdenum to ferritic Ductile Iron there is significant increases in creep and stress-rupture strength, which results in high temperature properties. [14]

2.1.7. 6. Effect of Manganese

It is a mild pearlite promoter which improves properties like hardness and proof stress to a small extent. It generally

- Retards the onset of the eutectoid transformation

- Decreases the rate of diffusion of carbon in ferrite
- Stabilizes cementite (Fe_3C).

Its can sometimes result in embrittlement[10, 14, 15]

2.1.7. 7. Effect of Copper

It is a strong pearlite promoter which increases the proof stress along with tensile strength and hardness. There is no embrittlement in matrix. In the pearlitic grade of the ductile iron the amount of copper is kept between 0.4 to 0.8% [10, 14, 15]

2.1.7. 8. Effect of Nickel

It helps in increasing the UTS (Ultimate Tensile Strength) without affecting the impact values. It is used in the range between 0.4 to 2.0%. As compared to silicon it also strengthens ferrite but is less effective in reducing ductility. It is also a mild pearlite promoter and increases proof stress but have little effect on tensile strength. It is very costly so it is present as traces in the matrix. [10, 14, 15]

2.2. Austempered Ductile Iron (ADI):

2.2.1. Austempering

In the early 1930's, the work of Bain, et al, on the isothermal transformation of steel developed the heat treatment process known as austempering process. In the early 1940's Flinn applied this process of austempering to cast iron, namely gray iron. In 1950's, both the austempering process and ductile iron had been developed.

In Austempering austenite transforms isothermally to lower bainite as a result it reduces distortion and cracking [16]. In this heat treatment process the steel is heated to a temperature uptoaustenitizing temperature and then the steel is quenched by holding the steel in a molten salt bath at a temperature above Ms and the Austenite is let to transform to lower bainite at this temperature.

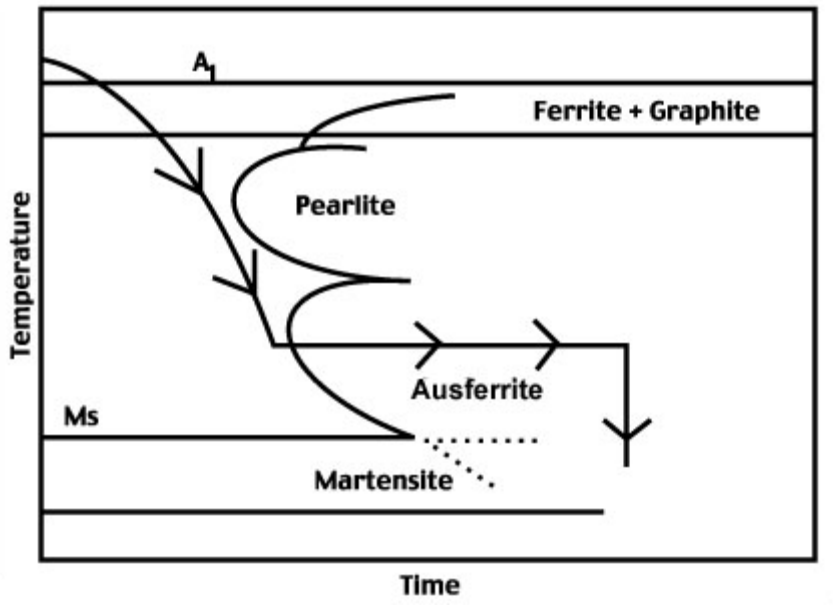

Fig5 .Austempering Process

When the steel is quenched from A1 temperature to the molten salt bath above Ms temperature, for proper austempering adequate hardenability of the steel is required to avoid pearlitic transformation. Also the bainitic transformation may be incomplete if the bainitic bay is long [16].

The process of Austempering results in

- enhanced ductility
- increased toughness
- higher hardness
- lesser distortion and quench cracks as compared with tempered specimen

ADI gives a combination of properties such as high strength with good ductility, good fatigue strength and wear resistancealong with fracture toughness [17]. Great combination of properties can be obtained by varying the Austempering variables i.e. austempering temperature and time of holding.

The microstructure of ADI consists of

- ferrite (α)
- High carbon austenite (γ_{HC}).

The microstructure of ADI is different from the austempered steels where the microstructure consists of ferrite and carbide. In ADI the product of austempering reaction iron is often referred to as "ausferrite" rather than bainite [17]. The addition of alloying element, silicon, suppresses the carbide precipitation during austempering reaction and retains high carbon austenite (γ_{HC}) [17,18]. In Austempering of steel the bainitic ferrite forms by the rejection of carbon into the residual austenite. As the process proceedsmore bainitic transformation takes place and more carbon is rejected into the surrounding austenite. This results in the increase of the amount of austenite and the amount of carbon in the austenite. In earlier stages, the austenite transform to martensite as the carbon content of austenite is insufficient to make it stable but at longer times it become thermally stable[19].

In the austempered ductile iron thebainitic transformation can be described as a two stage phase transformation reaction. In the initial transformation primary austenite (γ) decomposes to ferrite (α) and high carbon-enriched stable austenite (γ_{HC}). This transformation is commonly called stage I reaction [18].

Stage I: $\gamma \longrightarrow \alpha + \gamma_{HC}$

If the austempering time is too long, then a second reaction i.e. stage II takes place, where γ_{HC} decomposes into ferrite and carbide [18].

Stage II: $\gamma_{HC} \longrightarrow \alpha$ **+ Carbide**

Stage II reaction causes structure embrittlement and mechanical properties of ADI get degraded [18]. The carbide that is formed is ε carbide which makes the steel brittle [17].

2.2.2. Effect of copper:

Copper generally suppresses the carbide formation in lower bainite but do not alter the diffusion of carbon in austenite or its stability.[19]. It increases both the transformation rate and the carbon content in the matrix during austenitising process and widens theaustenite zone of the phase diagram. Dueto Cu addition stage II reaction is delayed and result in prevention from deterioration of properties. [20]

2.2.3. Microstructure:

With the progress of transformation during austempering, austenite transforms to bainitic ferrite. For short times, the formation of martensite cannot be prevented during cooling from

the austempered temperature to room temperature. Initially austenite is not sufficiently enriched with carbon to stabilize it but later onwith longer austemperingtimes,martensite disappears and the amount of bainitic ferrite and retained austenite increases. But at still higher austempering times, the amount of retain austenite decreases and this leads to lower ductility and impact energy values [20].

2.2.4.Production of ADI:

A typical composition of ductile iron casting used for making ADI is:

C 3.5% to 3.7%

Si 2.5% to 2.7%

Mn 0.25% to 0.31%

Cu 0.05% to 0.8%,

Ni 0.01% to 0.8%

Mo maximum of 0.25%

Austempered ductile iron produced by the austempering of ductile iron undergoes the following steps:

1. Austenitising: The ductile cast iron is heated to the austenitising temperature i.e. above A1 temperature (850˚C to 950˚C) so that the casting gets converted to austenite.

2. Austempering: The austenitised part is rapidly quenched in a molten salt bath to avoid pearlitic transformation. Quenching is done from the austenitising temperature of 850˚C - 950˚C in a salt bath maintained at a temperature of 200˚C - 450˚C.

3. Holding time: The casting is hold at the desired temperature for sufficient time to allow bainitic transformation to be complete.

4. Air cooling: After holding the sample for sufficient time it is air cooled to the room temperature.

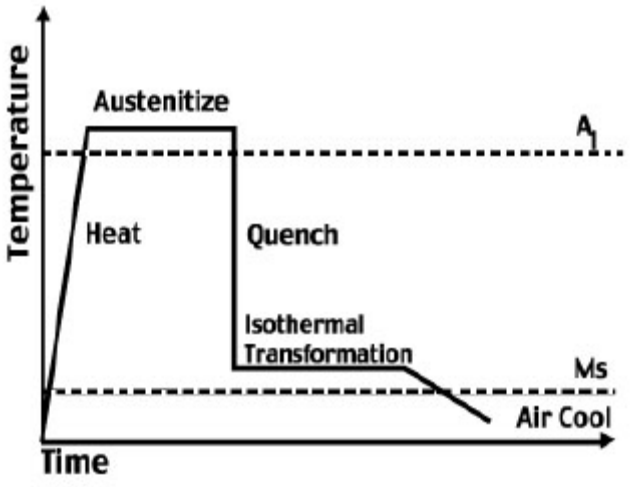

Fig 6. Schematic diagram for austemperingprocess

2.2.5 Unique properties of ADI:

The applications of ADI is very vast based on the properties coupled with cost and flexibility benefits in different sectors viz.

I. Agriculture: excellent resistance to soil wear.
II. Digger/grab teeth: high strength and wear resistance
III. Industrial: wear components,pumps etc.
IV. Gears: for wear resistance and better vibration damping than steel.
V. Construction: crushing,grading and wear components etc.
VI. Food and feeding milling: grinding,mixing, pelletisation etc.

Austempered Ductile iron has found successful application in many industries including construction and mining, automotive, heavy truck and rail road.

2.2.6. Disadvantage of ADI:

The major disadvantage of ADI is that welding is not recommended for this type of material.

CHAPTER 3

A Brief Discussion of Previous Work

A Brief Discussion of Previous Work

Many attempts were made to understand and predict the behaviours of austemperedductile iron that considers the study of aus-ferrite matrix structure and the response of the matrix structure to heat treatment, structure and properties correlation, and its mechanical properties with different variables and applications. A brief description of some of the literatures in these areas is presented here below.

Olivera Eric, DraganRajnovic, Slavica, LeposavaSidjanin, T. Jovanovicobserved and studied the microstructure and facture of two types of austempered ductileiron, one is alloyed with copper and another one is alloyed with copper and nickel. They also observed the effect ofcopper and copper plus nickel on the microstructure and impact properties of both the austempered ductile irons. According to them there is delay in the transformation kinetics of the residual austenite by the addition of copper plus nickel which results in a shift of the maximum of volume fraction of retained austenite to 3 hours of austempering,compared to 2 hours in austempered ductile iron alloyed with copper. In this way they could demonstrate that the volume fraction of retained austenite strongly effects the impact energy of both the irons, i.e., with content retained austenite up to maximum value impact energy increases, then a decrease occurs with the decrease retained austenite. [21]

Studies of J. Zimba, D.J. Simbi, E. Navara have shown the abrasive, wear and mechanical properties of the austempered ductile iron and comparison of these properties with that of the quenched and tempered steel. They found that as austempering temperature increases, the ferrite lath spacing and volume fraction of retained austenite increases. The tensile strength and hardness decreases with austempering temperatures while there is a significant increase in elongation and impact toughness as the austempering temperature is raised. The wear resistance of austempered ductile iron is good enough despite of the low initial hardness because during abrasion there is a surface transformation of retained austenite to martensite. Consequently the surface hardness and wear resistance of the iron is increased. [22]

Z.K. Fan and R.E. Smallman studied the fracture behaviour of the austempered aluminium spheroidal graphite iron. They took the specimens of aluminium spheroidal graphite iron containing by weight 3.2%C, 2.2%Al, 0.3%Si were austenitised at 950^{o}C for 2 hours and then austempered at 300^{o}C or 400^{o}C for a duration up to 6 hours, polished and squeezed to

fracture. From the observations they found that the cracks always originate from graphite nodules in austempered ductile iron. [23]

Studies of Uma Batra, S.Ray and S.R.Prabhakar shows the variation in the austempered microstructure, the volume fraction of retained austenite, the average carbon content of retained austenite, their product and the size of bainitic ferrite needles with austempering temperature for 0.6%Cu alloyed ductile iron. For this, copper alloyed ductile iron specimens were taken and austempered at different temperatures and times. They observed that the bainite morphology changes from lower bainite to upper bainite with increasing austempering temperature. Also, with increasing austempering temperature, the average volume fraction of austenite, its carbon content, and the size of bainitic ferrite increases. Increasing the austempering time initially increases the amount of retained austenite and its carbon content, and then both reach a plateau. The plateau extends over a period of stability of retained austenite, after which there is a decrease of both. [24]

Further thy studied the effect of alloying elements on the austemperingprocess, microstructure and structural parameters of both the austempered ductile iron containing 0.6% Cu and 0.6% Cu+ 1.0% Ni as the main alloying elements.Optical metallography and x-ray diffraction are used to study the changes of the structure. Effect of alloying additions on the austempering kinetics was studied using the Avramiequation.Theaustempering kinetics is slowed down by the addition of Ni. [11].

O. Eric, M.Jovanovic, L.Sidjanin and D.Rajnovic studied the microstructure and mechanical properties of the austempered ductile iron which is alloyed with Cu, Ni and Mo. When the austempering is done at 320^{o}C in the range of 2 to 5hours, a special type of austempered ductile iron microstructure is produced containing free bainitic ferrite and a stable highly carbon enriched retained austenite. During austempering of this iron at 400^{o}C; yield strength, tensile strength and ductility obtained are twice lower than that at 320^{o}C.The appearance of martensite in the microstructure pertains to the lower values of tensile properties. [25]

J. Aranzabal, I. Gutierrez, J.M. rodriguez-Ibabe and J.J. Urcolar studied the influence of the amount and morphology of austenite phase on the mechanical properties like proof stress, UTS and elongation and toughness at different austempering conditions of time and temperature. Then they concluded that the short time treatments lead to the deterioratedmechanical properties in accordance with the presence of untempered martensitic phase. [26]

Studies of A.S.M.A. Haseeb, Md. Aminul Islam, Md. Mohar Ali Bepari shows the behaviour of ductile iron heat treated through two different routes like quenching and tempering and austempering to equivalent matrix hardness. Under all test conditions, they observed, austempered ductile iron exhibits better wear resistance than quenched and tempered ductile iron, although both have similar chemical composition and matrix hardness. Micro-hardness measurement of the wear scratch reveals that the hardness of austempered ductile iron increases while that of quenched and tempered iron decreases during the wear process. [27]

M.NiliAhmadabadi, H.M.Ghasemi and M.Osia have studied the effect austempering process on the wear behaviour of the austempered ductile iron. They considered a 0.75 wt. %Mnductile ironwith different nodule counts and wasaustempered by conventional and successive austempering processes at 315^{o}C and 375^{o}C for different time periods. They conclude from the sliding wear tests on specimens with optimum mechanical properties austempered by different processes were delaminated which has a dominant wear mechanism .Thus the results from the mechanical and wear test shows that successive austempering processes improve both mechanical properties and wear resistance of ADI in comparison with conventional austempering process. [28]

Chapter 4

EXPERIMENTAL WORK

EXPERIMENTAL WORK

4.1. Experimental procedure:

Two grades of SG irons samples are collected and subjected to Austempering process for the present study. The chemistry of the received samples is as follows.

Table 2: Final chemistry of the receivedsample (wt %).

sample	C	Si	Mn	S	P	Cr	Ni	Mo	Cu	Mg
M1	3.68	2.22	0.16	0.011	0.026	0.02	0.03	0.02	0.003	0.048
M2	3.65	2.20	0.17	0.009	0.022	0.02	0.03	0.02	0.48	0.051

The received samples were cut and machined as per EN1563 specification for mechanical and metallurgical analysis in as-cast and after heat treatment is mentioned below.

4.2. Sample for mechanical properties analysis

Rounded specimen was made (14 mm dia and 70 mm gauge length) for tensile test. 10 samples were tested in servo-hydraulic machine (1000KN capacity) and another 10 samples also tested on the same way but after Austempering process.

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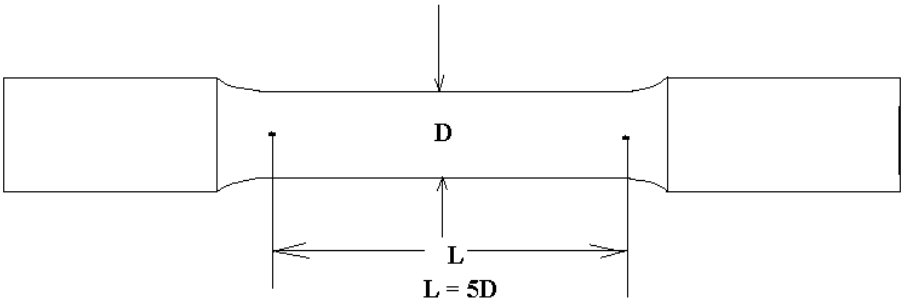

Fig 7: Schematic diagram of the rounded specimen for tensile test.

D = Gauge diameter = 14 mm.

L = Gauge length = 5x14 mm. = 70 mm.

For metallography analysis, samples were cut from the centre of the casting, ground and polished to get mirror polish. 2% Nital (2ml HNO_3+98ml CH_3OH) was used as etchant. All the specimen was undergone SEM analysis to characterize the graphite morphology and graphite distribution in the matrix.

4.3. Method adopted for Austempering

10 nos. of SG iron specimens of each melt were taken for carrying out austempering at four different temperatures (250^{o}C, 300^{o}C, 350^{o}C and 400^{o}C) for duration of 0.5 hour, 1 hour and 1.5 hour. Hardness values (RA) of ADI samples and their respective parameters are shown in figure 1. Samples were heated to 900^{0}C for one hour (austenitising) and then transferred quickly to a salt bath (four numbers) containing (50 wt% $NaNO_3$ + 50 wt% KNO_3) maintained at four austempering temperatures as mentioned for 0.5 hour,1 hour and 1.5 hour. Similarly another 10 nos. of specimens of each melt were made for tensile test. These specimens were inspected through radiographic test and the defect free specimens were subjected to heat treatment. The ADI test specimens were finally machined to their respective dimension as per EN1563 standard. Tensile tests were carried out with servo hydraulic UTM machine having 1000KN capacity. Results of tensile tests are shown in figure 2 and figure 3 respectively.

The main features of Universal Testing Machine is

I. rupture(%peak)=50
II. preload(%full scale)=0.05
III. safe load(KN)=900
IV. hold time(sec)=10
V. load rate(KN/min)=65
VI. stress rate(KN/sq.mm/min)=10
VII. elongation rate(mm/min)=2
VIII. strain rate(%strain/min)=1
IX. initial valve open(10-90%)=25

4.4.Hardness measurement

Samples are made 10mm×10mm×50mm dimension per hardness measurement. Samples were polished in SiC paper (fine polished) and hardness was measured in Vickers hardness tester. Average of 5 readings was taken for this purpose. This experiment was performed at room temperature and 10kg load was applied for 10 sec. The depth of the indentation was automatically recorded (diamond pyramid indenter) the diameter of each indentation was measured and R_A value was determined by a programming.

4.5. Scanning Electron Microscopy

The samples were made to 2mm thickness for this purpose. Fine polished was made and 2% Nital was used. The graphite morphology was characterized.

CHAPTER 5

Results and discussions

Results and discussions:

The mechanical properties and the metallography characterization has been studied in the present investigation. All the properties have been compared with Austempering variables. i.e. Austempering time and temperature. The results are discussed here under.

5.1. Effect of austempering time on hardness:

Table 3 and figure 8 list the hardness values of ADI specimen at different temperatures. Hardness values of M2 grade specimens are enhanced or increased as compared to M1 by 3 to 9 Rockwell hardness unit in A scale for different austempering conditions. This may be due to the large amount of pearlite present in the matrix of M2 specimens. Hardness is increasing from half an hour to one hour and then decreasing.

TABLE 3:

Effect of Austempering temperature and Time on Hardness (R_A) of ADI Samples.

Austenitising temp:850°C Austenitising time: 1 hr		Hardness Values(R_A)	
Austempering Temperature(°C)	Austempering Time(hr)	M 1	M 2
250	0.50	56	63
	1.00	64	73
	1.50	62	71
300	0.50	54	59
	1.00	63	69
	1.50	61	78
350	0.50	53	56
	1.00	61	67
	1.50	59	65
400	0.50	46	54
	1.00	56	63
	1.50	53	60

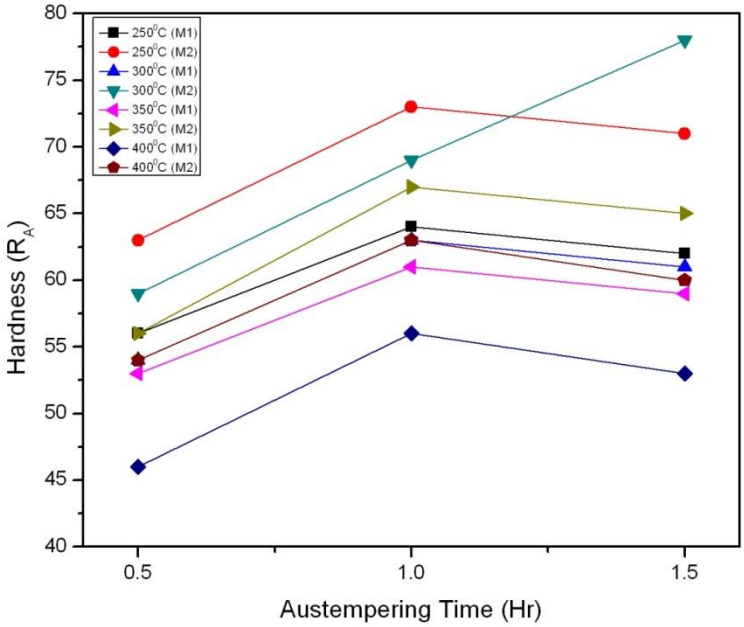

Fig. 8: Variation of Hardness with different austempering time of the melts M1 and M2 austempered at four austempering temperatures.

The alloyed ductile iron (alloyed with copper) gets increased as compared to specimens without copper as shown in the figure. Hardness is increasing from 30 min Austempering time to 60 min but from 60 min to 90 min it is decreasing. ADI process involve 2 stage mechanism viz. Stage 1 and stage 2.

In **stage 1:** $\gamma \rightarrow \alpha + \gamma_{HC}$

But carbon enrichment in retained austenite is too less to make the entire retained austenite stable at room temperature and some transformation to martensite is involved. With the increase in austempering time, the amount of retained austenite and bainitic ferrite increases until completion of bainitic transformation resulting in increase in hardness, tensile strength and yield strength. After completion of bainitic transformation, if austempering is continued for still longer duration, stage II reaction [2] sets in and retained austenite decomposes to bainitic ferrite and carbide.

Stage II: $\gamma_{HC} \rightarrow \alpha$ **+ carbide**

Stage II reaction is undesirable since it causes the embrittlement of structures and degrades the mechanical properties. This results in decrease of hardness, tensile strength and yield

strength after achieving a peak value [14].Hence as the Austempering time increases, the formation of Retained Austenite increases results in lower in hardness.

5.2. Effect of Austempering time on Tensile strength and yield strength:

Table 4, 5 Fig 9 and Fig10 lists the tensile strength values of ADI specimens at different austempering temperatures. From the results, it is observed that both tensile strength and yield strength is increasing from 0.5 hour austempering time to one hour and decreasing from one hour to 1.5 hour. ADI of M2 showing higher tensile strength as compared to ADI of M1.

TABLE 4.

Tensile properties of ADI Samples (M 1)

Aust. Temp(°C)	Aust. Time(hr)	UTS (Mpa)	Y.S (Mpa)	EL (%)
250	0.50	927	689	2.1
	1.00	1077	847	2.6
	1.50	1065	821	2.8
300	0.50	767	529	3.7
	1.00	921	686	4.3
	1.50	901	648	4.6
350	0.50	649	434	6.1
	1.00	809	561	6.9
	1.50	795	539	7.1
400	0.50	588	379	5.5
	1.00	766	521	5.8
	1.50	742	497	5.6

TABLE 5

Tensile Properties of ADI samples (M 2)

Aust. temp(°C)	Aust. time(hr)	UTS (Mpa)	Y.S (Mpa)	EL (%)
250	0.50	965	719	1.7
	1.00	1122	884	2.3
	1.50	1105	851	2.4
300	0.50	815	579	3.4
	1.00	973	732	4.1
	1.50	957	684	4.3
350	0.50	711	502	5.7
	1.00	869	634	6.4
	1.50	855	608	6.5
400	0.50	656	451	5.2
	1.00	815	592	5.6
	1.50	792	576	5.5

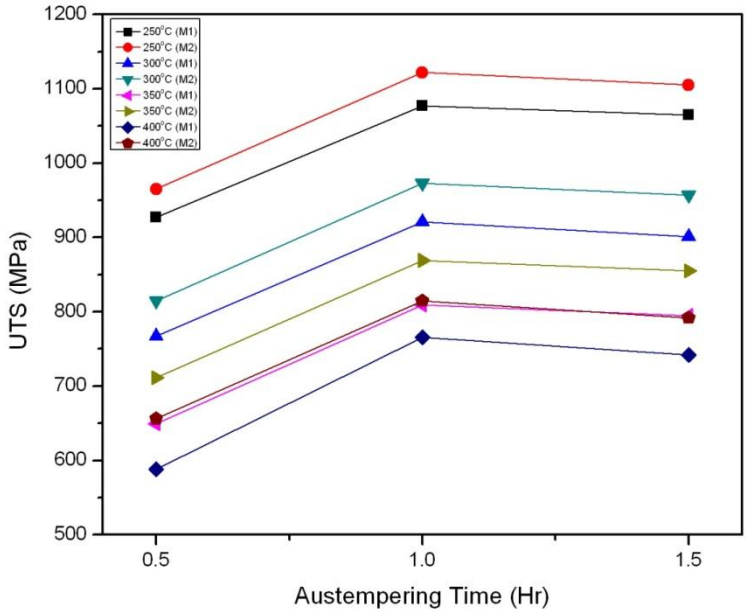

Fig. 9 Variation of Tensile Strength with different austempering time of the melts M1 and M2, austempered at four austempering temperatures

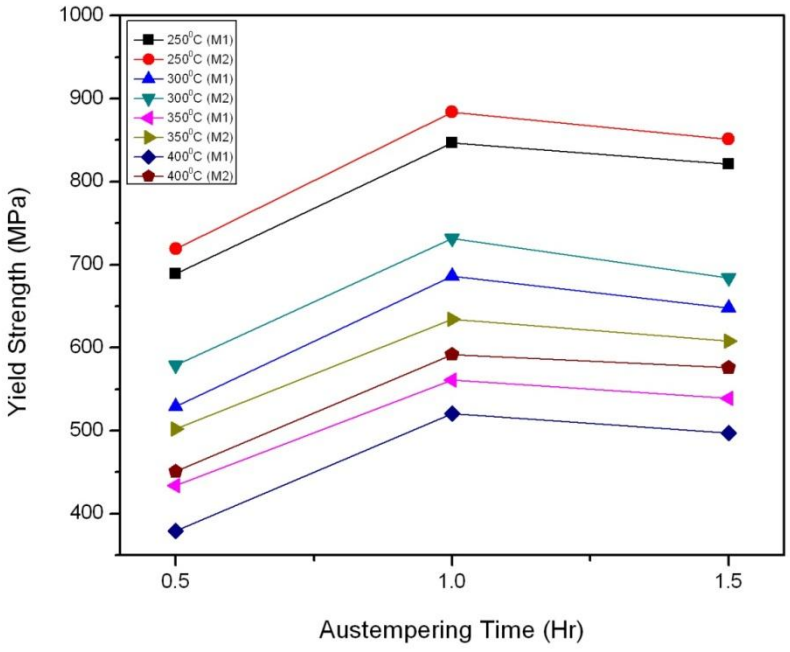

Fig. 10 Variation of Elongation with different austempering time of the melts M1 and M2, austempered at four austempering temperatures

The tensile properties (UTS and Y.S) of ADI specimen increases with increase in Austempering temperature. At low Austempering time high strength and high hardness values were achieved. This may be attributed due to the presence of acicular bainite and some percentage of martensite and retained austenite in the matrix. Low Austempering temperature accelerates the formation of fine bainite plates with non uniform distribution of carbides. Lattice distortion of the ferrite in the matrixand dislocation density may contribute for the property enhancement for the ADI. As the Austempering temperature increases, some percentage Retained Austenite and martensite is disappeared from the matrix which results results in lower values of strength and hardness. At higher Austempering temperature, coarser is are produced in lesser percentage leading to decrease in strength.

5.3. Effect of Austempering time on Elongation

Figure 5 lists the elongation values of ADI specimens at four different austempering temperature and time for both grades (M1 and M2). Elongation is increasing from half an hour austempering time to one hour, form one hour to one half hour it is increasing for all austempering temperatures (250^0C, 300^0C and 350^0C) but decreasing at 400^0C. ADI specimens of M 2 are showing lower elongation than ADI specimens of melt M 1.

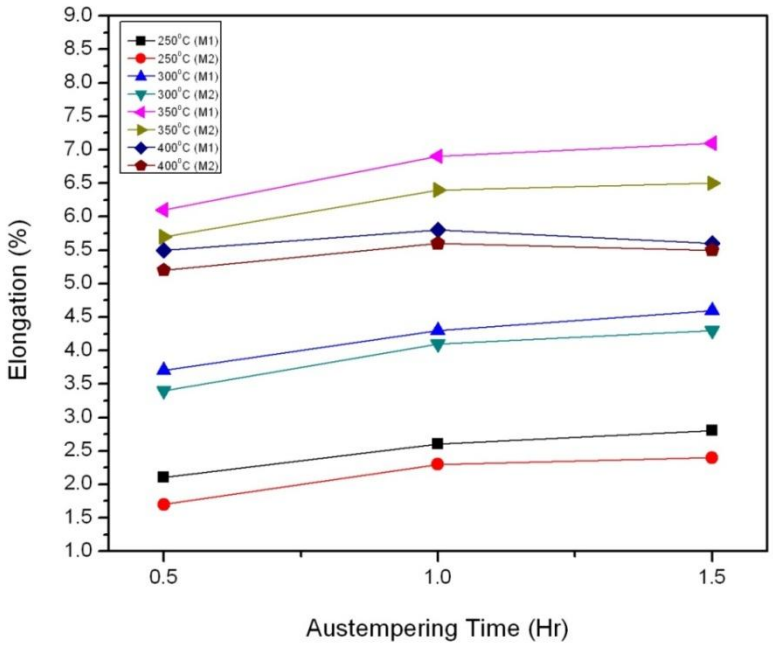

Fig. 11 Variation of Elongation with different austempering time of the melts M1 and M2, austempered at four austempering temperatures.

Low ductility of ADI is achieved at lower Austempering temperature. This may be attributed to brittle fracture which occurred in the matrix due to the presence of martensite. On the other hand the formation of retained austenite is less at lower Austempering temperature resulting decrease in ductility. The reverse is same for increase in Austempering temperature but after reaching a peak value (starting of stageII reaction) amount of retained austenite decreases as compared to lower Austempering temperature. This leads to decrease in ductility.

5.4. Characterization of graphite morphology:

The following figures shows the graphite morphology(isothermal transformation from austenite to bainite) is given at different Austempering temperature.

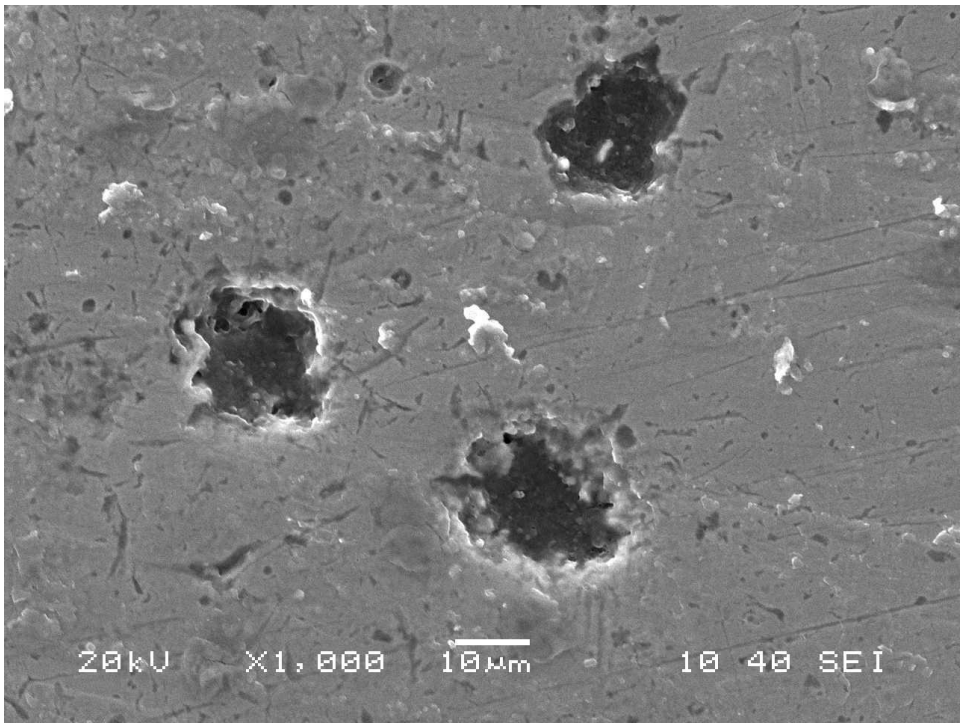

Fig.12 Micrograph of ADI austempered at 250^{o}C for 60 min(M1)

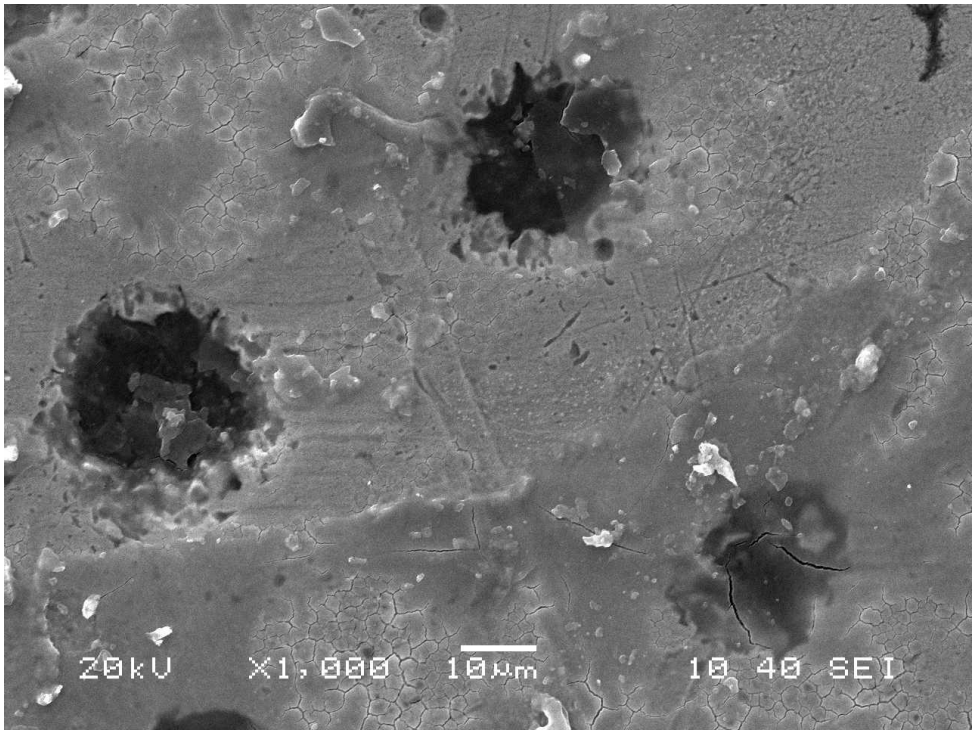

Fig.13 Micrograph of ADI austempered at 250^{o}C for 90 min(M1)

The above microstructure shows lower bainite structure which has been austempered at higher austempering temperature. Graphites are swallowed in the matrix as it is observed in the figure. Retained austenite arenon uniformly distributed in the matrix.This may be formation of lower bainite and small volume fraction of austenite (Fig 13) which is present in the bainiticferritic needles. Shallow dimples are observed and may be due to the presence of lower bainite and lower quantity of retained austenite in the microstructure.

The low ductility for shorter austempering times (Fig 12 and 14) can be attributed to some brittle fracture taking place due to the presence of martensite in the microstructure (Jung et al., 2005, Batra et al., 2004). But with increasing austempering time, the amount of retained austenite increases resulting in increase of elongation (Fig 13). This reaches maximum at the completion of stage I reaction and with the onset of stage II reaction. The ductility decreases owing to the decrease in retained austenite. Moreover the amount of retained austenite at low temperature is less resulting in lesser elongation.

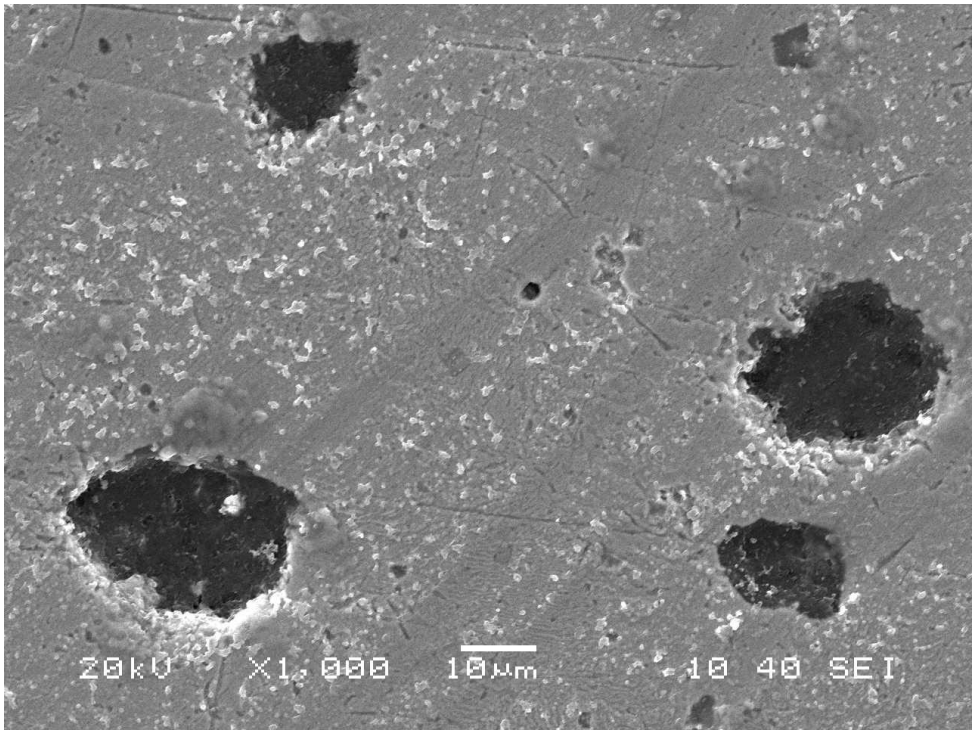

Fig.14 Micrograph of ADI austempered at 300^{o}C for 60 min(M1)

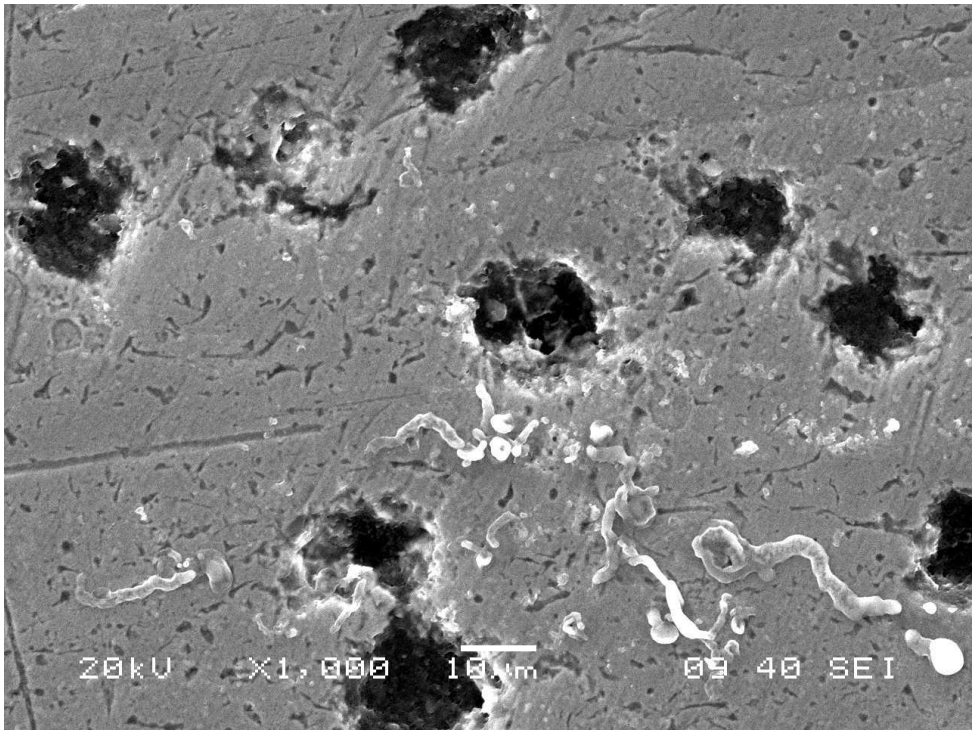

Fig.15 Micrograph of ADI austempered at 300^{o}C for 90 min(M1)

As the austempering temperature increases, the morphology of bainite is changed from acicular to plate like structures. The percentage of retained austenite is increasing at higher austempering temperature. As strength is lower at lower austempering temperature there is no

significant difference between M1 and M2 grade of spheroidal graphite iron.With the increasing austempering temperature, the amount of retained austenite increases and the ductility decreases. But after reaching some maximum elongation, at still higher austempering temperature the stage II reaction is more pronounced and proceeds at a faster rate than that at lower austempering temperature. Thus, this leads to some decrease in ductility. The hardness, tensile strength and yield strength of ADI specimen decreases with increase in temperature but the ductility initially increases with temperature and then after reaching a peak value it starts decreasing. At lower austempering time, the high strength and high hardness value can be attributed to the presence of acicular bainite, some martensite and retained austenite (Ali et al., 2000; Hsu and Lin, 2011). The fine structure of the bainite plates and low amount of retained austenite results in high strength at low austempering temperature. At higher austempering temperatures (at 350^0C and 400^0C), it is observed that the dimples become deeper and more numerous. It may be due to the presence of upper bainite and large volume fraction of austenite.Deep dimples and river like features in a few places are found and this may be attributed to martensite at the centre of the retained austenite (Shelton and Bonner, 2006; Putatunda,2001).

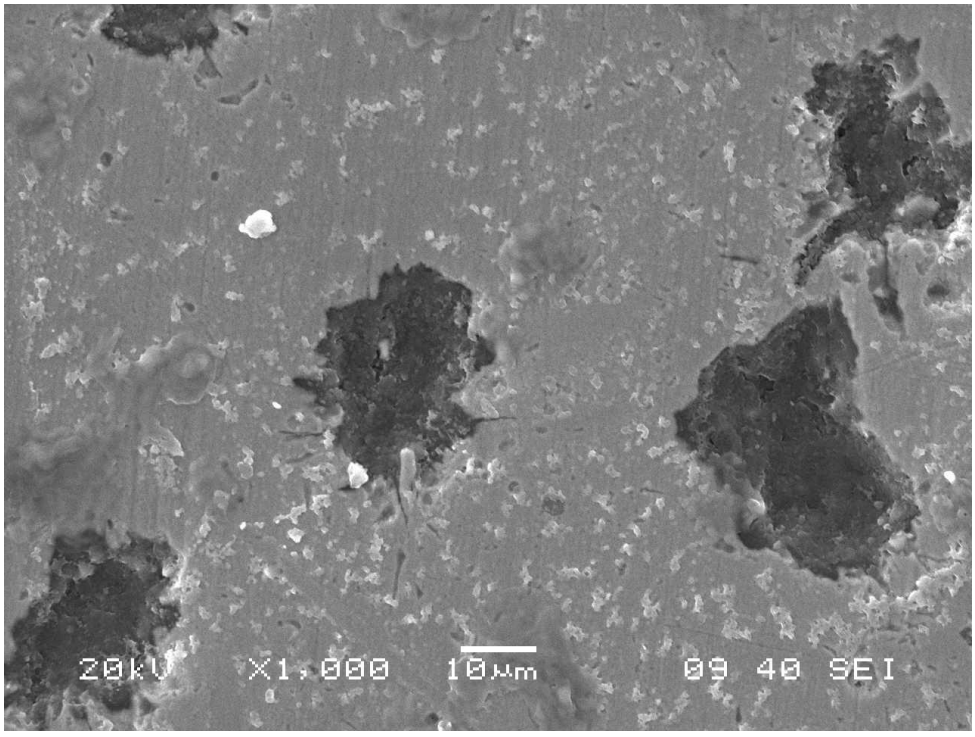

Fig.16 Micrograph of ADI austempered at 350^oC for 60 min(M2)

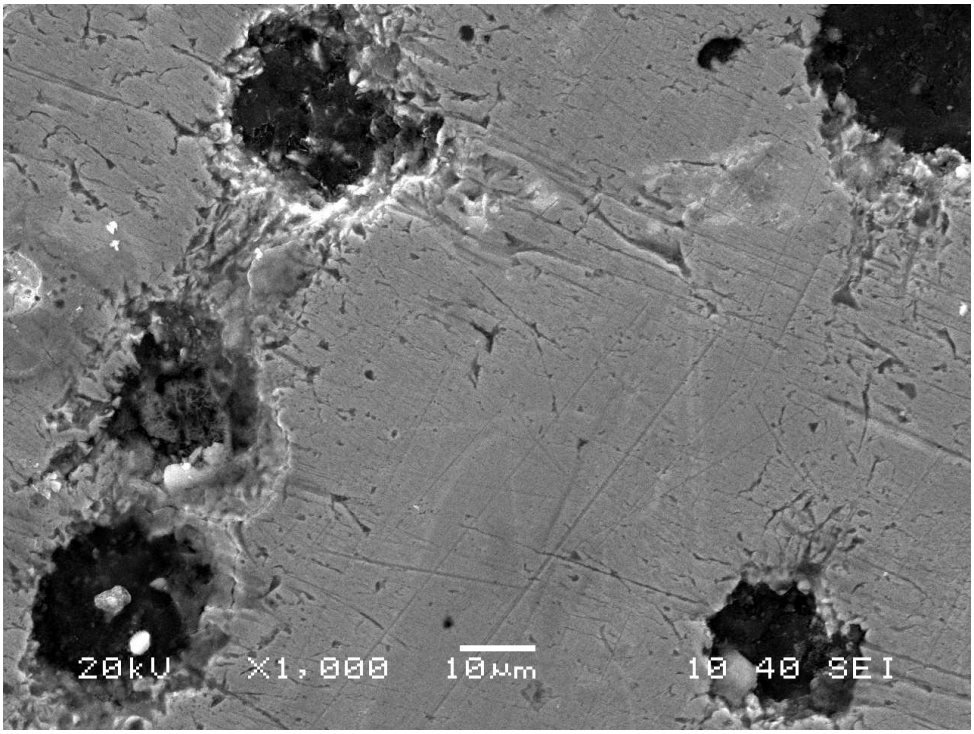

Fig. 17 Micrograph of ADI austempered at 350^{o}C for 90 min(M2)

In the above two pictures it is found that retained austenite and some percentage of martensite is present is due to dimpled fracture. River like structures are found in some cases. It is observed that dimple size and no. of dimples both are increasing with increase in austempering temperature. This indicates there is improvement in ductility.Moreover, other factors such as dispersed carbides, high dislocation density and lattice distortion of the ferrite contribute to the mechanical properties.

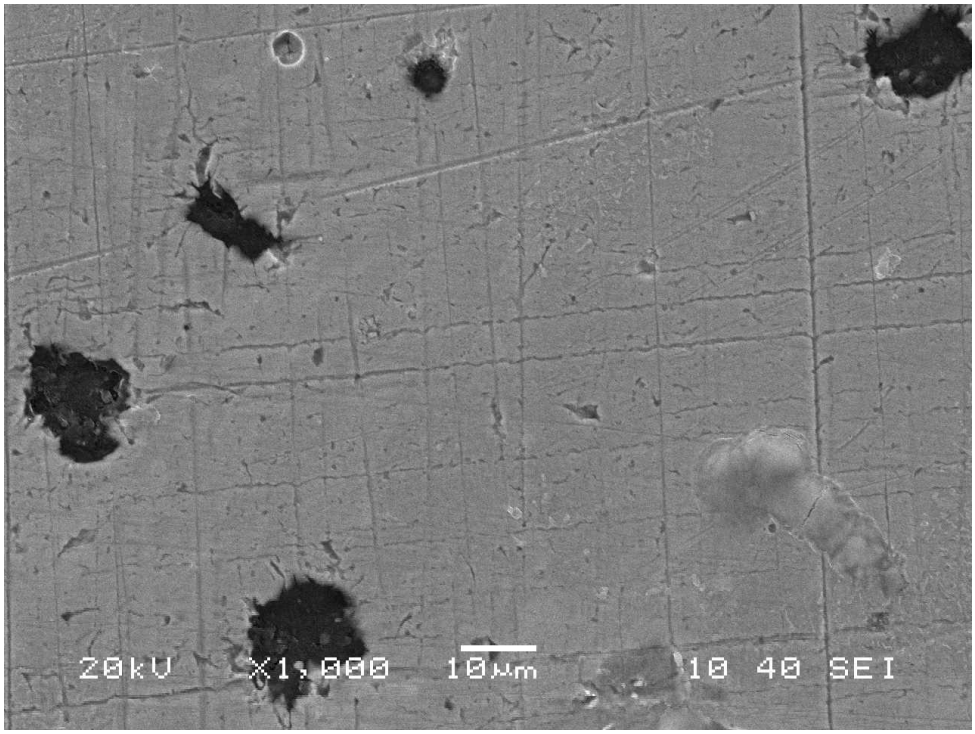

Fig.18 Micrograph of ADI austempered at 400^{o}C for 60 min(M2)

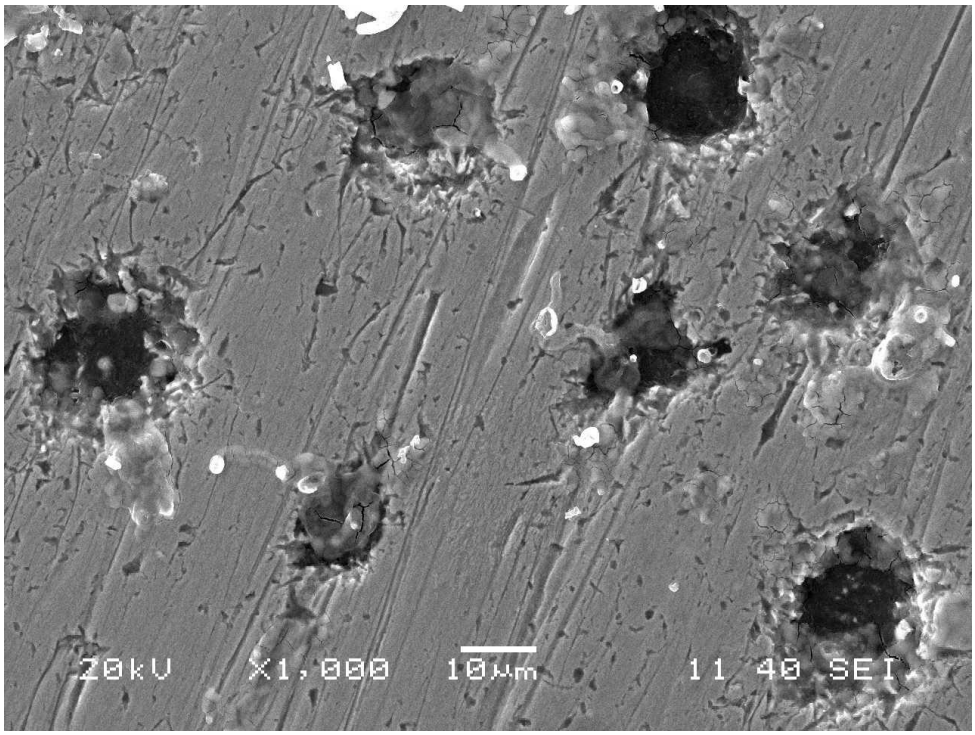

Fig.19 Micrograph of ADI austempered at 400^{o}C for 90 min(M2)

With the increase in austempering temperature, the amount of retained austenite increases and martensite disappears from the microstructure resulting in decrease in strength and hardness (Batra et al., 2000, 2003). At higher austempering temperature, bainitic ferrite produced is coarser but in lesser volume, leading to decrease in strength.

CHAPTER 6

CONCLUSIONS

Conclusions:

The effect of alloying element (Cu) on the mechanical properties of SG iron austempered at four different temperatures with varying austempering time has been investigated in the present investigation. The following conclusions are made:

1. Alloying element (Cu) improves the mechanical properties of spheroidal graphite iron after austempering.The propertiesareenhanced with austempering time but with increasing austempering temperature it initially increases and then gradually becomes constant.

2. The ductility of ADI also initially increases with austempering time up to a certain value and then it starts decreasing with further increase in time.Hardness,tensile strength and yield strength of ADI decreases continuously with austempering temperature.

3. The ductility of ADI initially increases with austempering temperature and then after reaching some maximum value at around 350^{o}C, it starts decreasing with further rise in temperature.

6.1.Future scope of work

Many works may be required to properly characterize both mechanical properties as well as graphite morphology of Austempered Ductile Iron. DSC analysis should be carried out at every austempering time and temperature to justify the transformations in different conditions in the matrix. XRD analysis should be carried out in the diffraction angle range from 50degree to 120degree for clear understanding of the lattice, lattice plane and different phases. Austempering time and temperature may be extended further to observe the change in mechanicalbehaviour.

CHAPTER 7

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